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Removal of heavy metals from industrial waste solutions by a rotating fixed bed of ion exchange resin

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ABSTRACT

The rate of removal of copper ion (Cu^{2+}) from industrial waste solution was studied using a rotating fixed bed of cation exchange resin. Variables studied were initial concentration of Cu^{2+}, pH value, rotational speed, and ratio of mass of resin to the liquid volume (S/l). Best removal capacity was obtained in the pH range (4–5). The experimental results revealed that the rate of removal of Cu^{2+-} decreases as the initial concentration of Cu^{2+} increases, while the rate increases with increasing pH value, contact time, rotational speed, and the mass of resin per unit volume of solution. The results were treated mathematically by using the method of dimensional analysis and mass transfer correlation was obtained. Langmuir and Freundlich isotherms were examined where Langmuir adsorption isotherm gives a better data fitting than Freundlich isotherm

Keywords: Adsorption; Ion exchange; wastewater; Heavy metals; Copper ion; Mass transfer; Rotating packed bed reactor

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1. Introduction

Discharge of industrial waste water containing toxic heavy metals such as Cu^{2+}, Cd^{2+}, Hg^{2+}, Cr^{6+} poses serious threat to the environment because these pollutants are nonbiodegradable and tend to accumulate in living organisms and lead to fatal diseases. In case of wastewater containing Cu^{2+}, wastewater treatment not only rid water of a toxic metal ion but also recovers a precious metal. Copper ions are discharged from industries such as printed circuit industry, mining industry, electrowinning, electrorefining of copper, metal finishing industry which include electroplating and electroless plating of copper, pickling and etching of copper and copper alloys, electropolshing of copper and copper alloys, electroforming of copper articles and electrochemical machining of copper alloys.

Different techniques have been used for copper removal from waste solutions such as electrodeposition, cementation and liquid- liquid extraction, membrane separation, and ion exchange resin. For extremely dilute solutions of Cu^{2+} the ion exchange method seems to be more suitable than other methods [1]. Also different types of low cost adsorbents have been examined for copper ions removal from waste solutions for example saw dust, iron oxides [2], wheat shell [3], bagasse fly ash [4] and also spent activated clay [5].

Previous studies on the use of ion exchange resins for heavy metal removal have used either fluidized beds or fixed beds of the resin, both techniques suffer from drawbacks such as the high pressure drop in case of fixed beds and the low slip velocity in case of fluidized beds, the low slip velocity leads to a low rate of mass transfer between the solution and the resin particles with a consequent low rate of heavy metal removal [1,6,7].

The aim of the present work is to explore the possibility of using a rotating fixed bed of ion exchange resin for copper removal, the ion exchange particles are packed in circular basket made of stainless steel screen. Basket rotation

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would enhance the rate of mass transfer of the Cu^{2+} ions to the resin particles by virtue of the flow induced inside the bed by the centrifugal force [8].

2. Experimental part

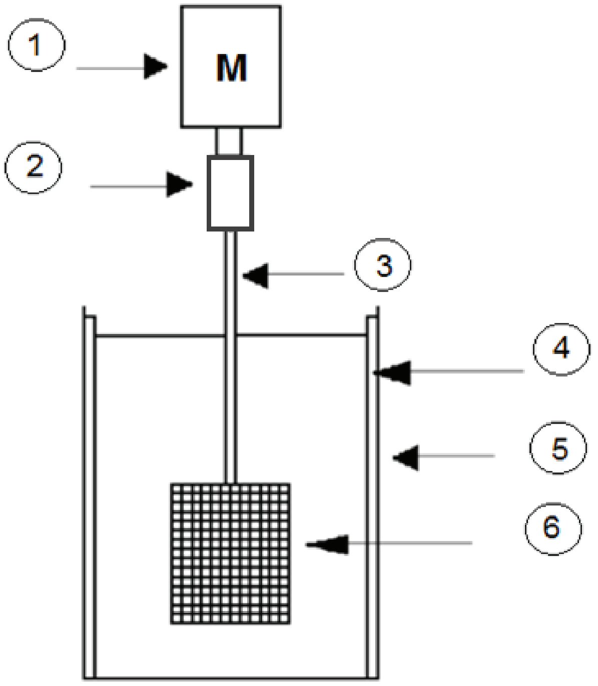
2.1. Apparatus

The experimental set-up used in the present work is shown in Fig. 1. It consisted mainly of 4.5 L plexiglass vessel with 25 cm height and 18 cm diameter. Four baffles were fixed on the wall of the vessel at 90°, baffle dimensions were: length = 25cm, width = 1.5 cm [9]. A cylindrical basket of 8 cm height and 6 cm diameter made of stainless steel screen with mesh no. of 40 was used. The cylindrical basket was covered tightly from its top by a screen disc cover to prevent the resin from escaping out. The basket was connected to a shaft of 0.5 cm diameter which was connected to a variable speed dc motor of 0.5 hp. Motor rotation speed was controlled by means of a variac and was measured by an optical tachometer.

2.2. Materials used

2.2.1. Cation exchange resin

A strong cation exchange resin, macroporous MonoPlus SP 112 (Laxness energizing chemistry) in the sodium form was used in the present work.



1- Electric Motor	4- Baffles
2- Plastic sleeve	5- Cylindrical plexiglass
3- Rotating shaft	6- Stainless steel screen basket

Fig. 1. Schematic diagram of the batch experimental setup.

Before using the resin, it was treated with a solution of sodium hydroxide. The treatment step was carried out by immersing the resin in sodium hydroxide solution of 1.5 M concentration for 45 min to ensure that all the resin is in the sodium form, and the following steps were carried out to complete the pretreatment step [10]. The resin was washed with distilled water three times to remove excess OH^- group. The following processes were carried out on the resin respectively:

A. Immersing the resin in 2 M HCl solution for 45 min to replace all sodium ions by hydrogen ions.
B. Washing with distilled water three times to remove excess Cl^- ions.
C. Drying at ambient temperature for 2 days.
D. Drying in a drying oven at 60°C for 20 min.

2.3. Procedure

Before each run, the cylindrical plexiglass container and the stainless steel basket Fig. 1, were washed with tap water, distilled water and dried. Before each run 4.5 L of fresh copper sulfate solution of the required copper ion concentration were placed into the cylindrical vessel. The initial copper ion concentration used in the present work were 300, 400, 500 ppm respectively. The required amount of treated resin were placed into the cylindrical basket namely 3 g, 4 g, 5 g per 1 L of solution respectively. The basket which was connected to the motor shaft was completely immersed in the solution; the rotational speed was adjusted by the variac and was measured by optical tachometer. The rotational speed used were 100, 200, 300, 400, 500, 600 rpm. Kinetics of the ion exchange reaction was followed by withdrawing 5 ml sample from the vessel every 2 min, the samples were analyzed for the remaining copper ion concentration by iodomertric titration [11].

All experiments were carried out at 25°C. Density and viscosity of the solution used were determined using a density bottle and an Ostwald viscometer respectively [6]. The diffusivity of copper ion in solution was obtained from the literature and was corrected for the change in temperature using the Stokes-Einstein correlation [12,21].

$$D\mu/T = \text{constant} \qquad (1)$$

3. Results and discussion

3.1 Mass transfer analysis and data correlation

There are two mass transfer resistances in ion exchange reactions; the first is the external mass transfer resistance due to the liquid film or boundary layer surrounding the ion exchange particle. The second is the intra-particle diffusion resistance due to the porosity (ion diffusion inside the resin particle). Either one of them or both resistances can be the rate-controlling step [7,13,14].

Liquid phase diffusion is sensitive to stirring while intraparticle diffusion is not sensitive to stirring [15]. Assuming a pseudo first order reaction, the rate of metal ion removal by the ion exchange resin in a batch reactor is given by [16].

$$V\,(dC/dt) = \mathrm{KAC} \qquad (2)$$

which upon integration for the conditions $t = 0$, $C = C_o$ & at $t = t$, C=C yeilds

$$\ln (C_o/C) = (\mathrm{KA}/V)\, t \qquad (3)$$

where $C_{o:}$ initial concentration of copper ion, ppm; C: concentration of copper ion at any time, ppm; V: solution volume in cm^3; t: time of reaction in s; K: rate constant in cm/s; A: surface area of resin in cm^2. It was calculated from the following equation:

$$\mathrm{A} = (6\ \mathrm{W})/\ (d_p\rho) \qquad (4)$$

where W: mass of resin in g; d_p: average diameter of resin in cm; ρ: density of resin in g/cm^3.

Fig. 2 shows a typical plot of ln (C_o/C) vs. time at different rotational speeds, the data fit Eq. (3), this suggests that the reaction is a first order diffusion controlled reaction in view of the sensitivity of the rate of the reaction to bed rotation speed [16].

A plot of ln (C_o/C) vs. t gives a straight line of slope (KA/V). The mass transfer coefficient of Cu^{2+} removal by the resin was obtained from the slope (KA/V) under different operating conditions.

Fig. 3 shows the effect of speed of rotation on the rate constant K at different doses of resin in g/L of solution.

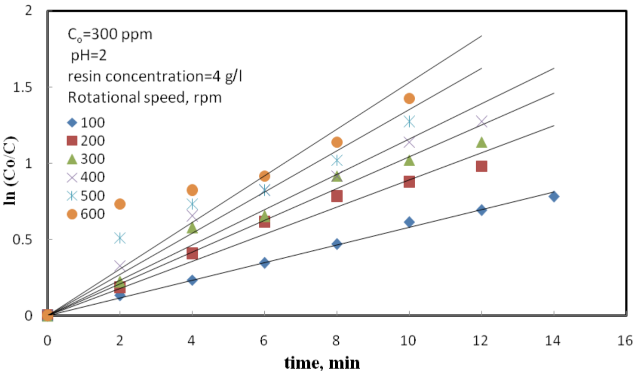


Fig. 2. ln (C_o/C) versus contact time at different rotational speed.

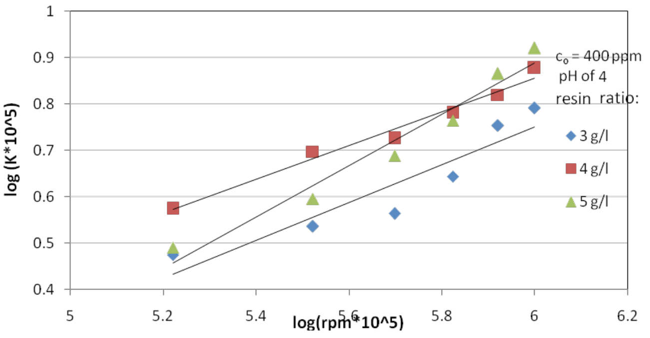


Fig. 3. Effect of rotational speed on the rate constant (K) at different mass of resin per liter of solution.

Fig. 4 shows that the degree of Cu^{2+} removal increases with increasing bed rotation speed and reaches a maximum of 88% .

The data fit the following equation:

$$\mathrm{K} = \mathrm{a\ N}^{\,0.514} \qquad (5)$$

The increase in the mass transfer coefficient K with increasing bed rotation speed can be explained in terms of the flow pattern at the rotating fixed beds. According to Bisang et al. [8] when the solution inside the bed moves radially by the centrifugal force, it is replaced by fresh solution which moves axially towards the bed (Fig. 5).

As the solution moves radially and axially past the ion exchange particles a diffusion layer of thickness δ is built around each particle the thickness of which decreases with increasing N with a consequent increase in K according to the equation

$$\mathrm{K} = \mathrm{D}/\delta \qquad (6)$$

The increase in the rate of Cu^{2+} removal with increasing the degree of stirring suggests that the liquid phase dif-

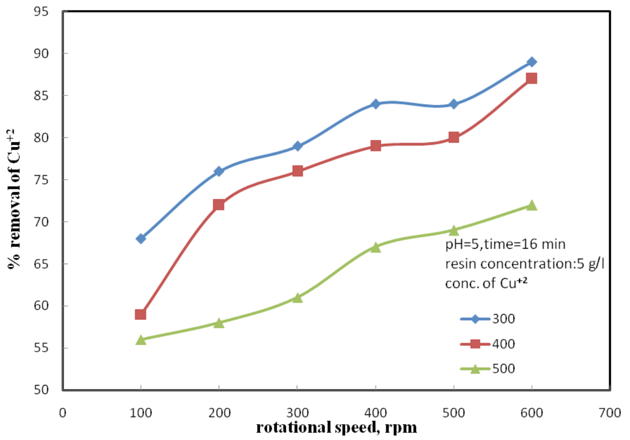


Fig. 4. Effect of speed of rotation on the percentage removal of Cu^{2+} at different initial concentration of Cu^{2+}.

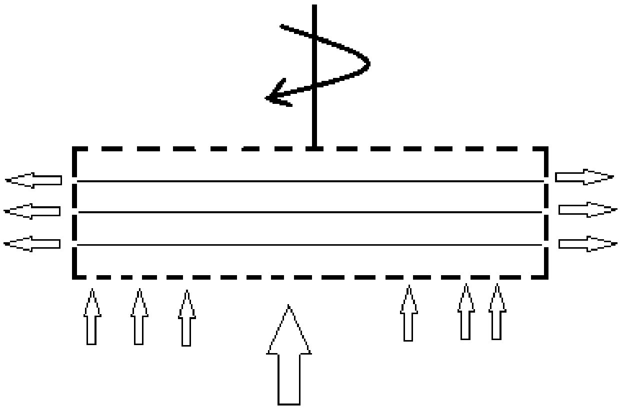


Fig. 5. Approximate flow pattern at the rotating basket.

fusion of Cu^{2+} across the diffusion layer surrounding each particle (Fig. 6) is the rate determining step given the fact that intraparticle diffusion is not sensitive to stirring [16].

The exponent 0.514 is in agreement with the value 0.5 obtained for diffusion controlled reactions at a solid rotating disc under laminar flow conditions [17,18].

The data were correlated in terms of the dimensionless groups Re, Sh, and Sc usually used in correlating forced convection mass transfer data.

$$Re = \rho N d^2/\mu \qquad (7)$$

where N = rotational speed (rps)

$$Sh = Kd/D \qquad (8)$$

$$Sc = \mu\rho/D \qquad (9)$$

Fig. 7 shows the relation between log Sh versus log Re at different Sc number, the data fit the Eq. (10)

$$Sh \; \alpha \; Re^{0.523} \qquad (10)$$

Fig. 8 shows that the present data for the conditions:

$1312 < Sc < 1403$, $20687 < Re < 118702$, fit the overall mass transfer Eq. (11):

$$Sh = 1.235{*}Sc^{0.33}\,Re^{0.523} \qquad (11)$$

with an average deviation of ±9 %

Many overlapping points close to the straight line were eliminated from Fig. 8 for the sake of clarity.

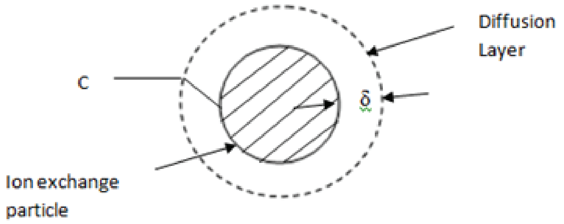


Fig. 6. Formation of a diffusion layer around each particle and Cu^{++} concentration inside the diffusion layer.

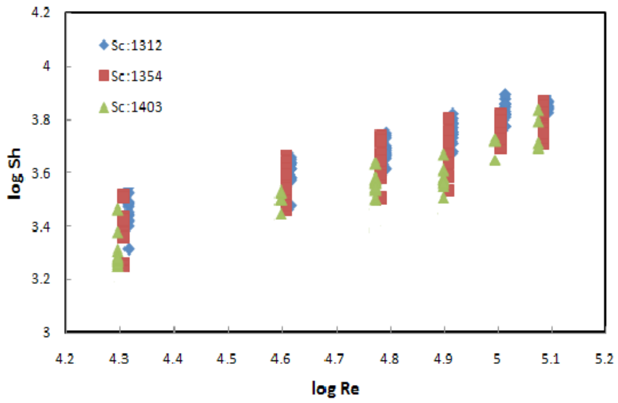


Fig. 7. Log Sh versus log Re at different Sc values.

3.2. Factors affecting the % Cu^{2+} recovery

3.2.1. Effect of initial Cu^{2+} concentration

Fig. 4 shows the effect of initial Cu^{2+}concentrations on the percentage removal of Cu^{2+}, the data show that the percentage removal of Cu^{2+} decreases as the initial Cu^{2+}concentration increases from 300 to 500 ppm. This result may be explained by the fact that for a given amount of resin, as the initial concentration of Cu^{2+} increases, the limited number of active sites of the resin available for Cu^{2+} adsorption will be exhausted, any further increase of Cu^{2+}concentration will remain unadsorbed by the saturated resin.

The present result is in agreement with the finding of Veli et al. [19] who studied Cu^{2+} removal from aqueous solutions by ion exchange resin [Dowex HCR S/S and Dowex Marathon C] using a fluidized bed in agitated batch reactor.

3.2.2. Effect of contact time

Fig. 9 shows the effect of time on the percentage uptake of Cu^{2+} at different initial Cu^{2+} concentrations and different doses of resin in gram per liter of solution. The data show that the %Cu recovery increases with increasing the contact time and reaches the equilibrium value after 16 min, the percent copper recovery at equilibrium ranges from 39% to 69% depending on the initial copper ion concentration, the

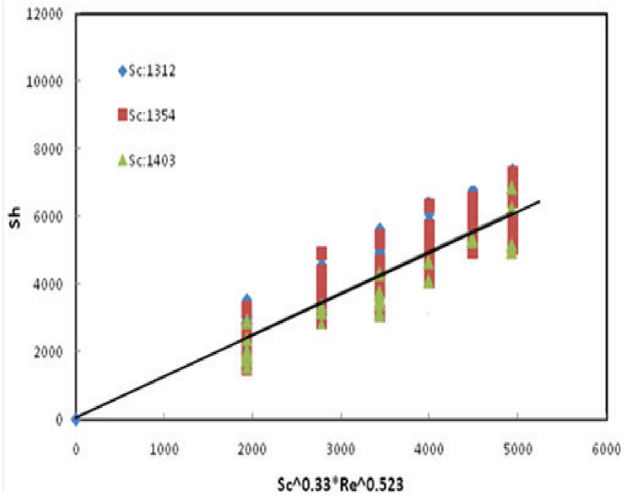


Fig. 8. Overall mass transfer correlation.

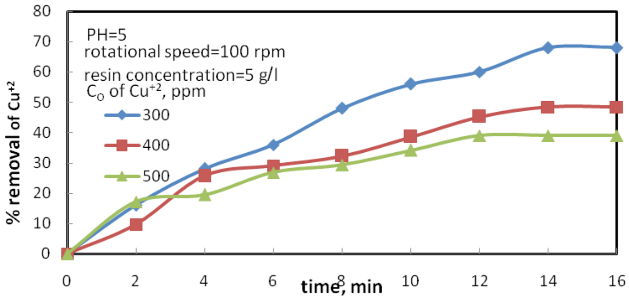


Fig. 9. Effect of contact time on percentage removal of Cu^{2+} at different initial concentration of Cu^{2+}.

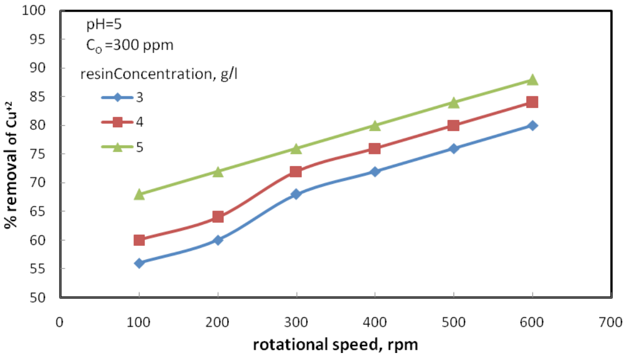


Fig. 10. Effect of rpm on percentage of Cu^{2+}removal at different mass of resin concentration.

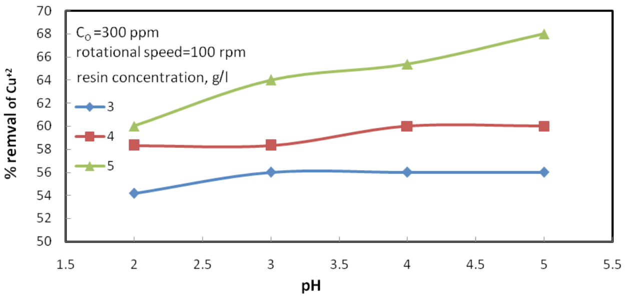


Fig. 11. Effect of pH on the percentage removal of Cu^{2+} at different mass of resin per liter of solution.

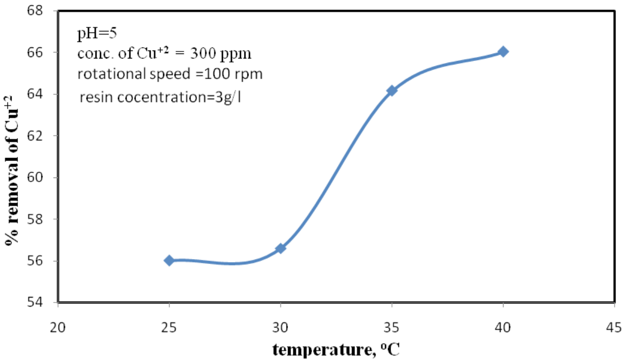


Fig. 12. Effect of temperature on percentage removal of Cu^{2+}.

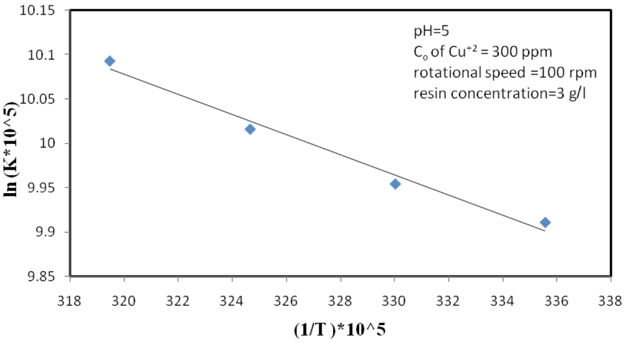


Fig. 13. ln K. versus 1/T at different temperature.

higher the initial Cu^{2+} concentration, the higher the % equilibrium removal.

The present result is in agreement with the results of Dave et al. [20] who studied the removal of Cu^{2+} by a fluidized bed of anion exchange resin (Dorex), the authors found that as the contact time increases, the percentage removal of Cu^{2+} increases.

3.2.3. Effect of the amount of resin

Fig. 10 shows the effect of the amount of resin in gram of resin per liter of solution on the percentage removal of Cu^{2+}. The data show that as the amount of resin increases, the percentage of Cu^{2+} removal increases. At a fixed speed of rotation of 600 rpm the percentage removal of Cu^{2+} increased from 58% to 87% when the amount of resin increased from 2 to 3 mg/L.

It is well known that the ratio of the number of available exchange sites to the number of Cu^{2+} increases with increasing the amount of resin, this results in an increase in the percentage removal of Cu^{2+} the present results are consistent with the results of Dave [20].

3.2.4. Effect of solution pH

Fig. 11 represents the effect of initial pH on the percentage removal of Cu^{2+}. Increasing the pH from 2 to 5 increases the percentage uptake of Cu^{2+} from (55% to 68%). At pH higher than 5, the adsorption of Cu^{2+} decreases probably because of the precipitation of Cu^{2+} as copper hydroxide around each particle thus isolating the resin particles from the solution.

3.2.5. Effect of temperature

The effect of temperature was studied at rotational speed of 100 rpm, initial concentration of Cu^{2+} 300 ppm, amount of resin 3 g/L of solution, and pH of 5. As shown in Fig. 12, it can be noticed that as temperature increases from 25 to 40°C, % Cu^{2+} removal increases from 56% to 66%. This may be attributed to the fact that as temperature increases, the viscosity of the solution decreases with a consequent increase of Cu^{2+} diffusivity according to Stokes-Einstein equation [Eq. (1)] [21].

The present results are in agreement with the results of Dave et al. [20] who found that as temperature increases, the percentage removal of Cu^{2+} increases.

In order to confirm that the uptake of Cu^{2+} by the resin used in the present study is diffusion controlled reaction, a plot of ln K vs. (1/T) was carried out according to Arrhenius equation [22]:

$$K = A*e^{-E/(R*T)} \qquad (12)$$

E was calculated from Fig. 13 and was found to be 2.1 kcal/mol.

The relatively low value of the activation energy (<5 kcal/mol) confirms the diffusion controlled nature of the present reaction [23].

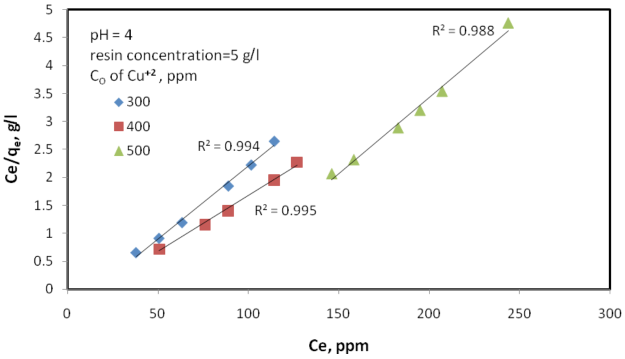


Fig. 14. Plot of Langmuir adsorption isotherm.

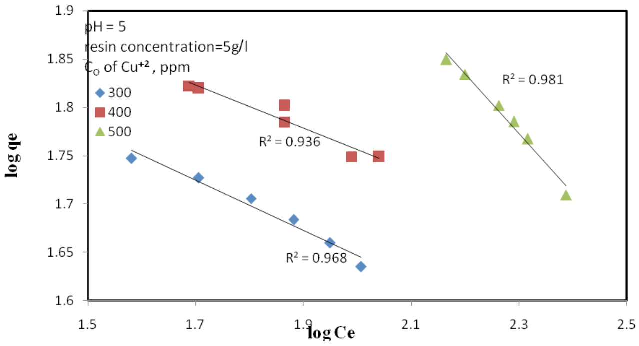


Fig. 15. Plot of Freundlich adsorption isotherm.

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Table 1
Range of different parameters studied in the present work

Parameter	Range
Initial concentration of Copper ion, ppm	300–500
pH	2–5
Speed of rotation, rpm	100–600
Temperature, °C	25–40
Mass of resin per liter of solution, g/l	3–5
Contact time, min	0–20

Table 2
Isotherm constants for Cu^{2+} sorption in ion exchange resin MonoPlus SP 112 for initial concentration of Cu^{2+} of 400 ppm

Isotherm model	Parameters	pH = 4		
		Dose of resin		
		3 g/l	4 g/l	5 g/l
Freundlich	K_F	7870.458	319.89	193.1968
	$1/n_F$	–0.884	–0.314	–0.252
	R²	0.996	0.952	0.975
	X^2	3.485	2.658	1.895
Langmuir	Q_m	34.483	40.000	37.037
	K_L	–0.006	–0.011	–0.013
	R²	0.996	0.992	0.995
	X^2	1.626	1.023	0.838

3.3. Isotherm studies

The adsorption capacity after reaching equilibrium, q_e (mg/g), was calculated using the formula [24,25]

$$q_e = (C_o - C_e)V/W \tag{13}$$

where C_o and C_e (mg/l) are the initial and equilibrium concentrations of Cu^{2+} respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent (g).

It is important to evaluate the most appropriate isotherm which fit the present data to optimize the design of a sorption system. Langmuir, Freundlich isotherm models were used to describe the adsorption equilibrium. Experimental isotherm data were conducted at an equilibrium time of 16 min for different amount of resin per liter of solution, different initial concentration of Cu^{2+}, and different pH value.

3.3.1. Langmuir model

The Langmuir adsorption isotherm Fig. 14 is based on the assumption that all sites possess equal affinity for the adsorbate. It may be represented in the linear form as follows:

$$C_e/q_e = 1/(K_L{*}Q_m) + (C_e/Q_m) \tag{14}$$

Using the present data the constants K_L and Q_m were determined and found to be:

$$K_L = -0.06 \text{ L/mg} \quad Q_m = 38.5 \text{ g/L}$$

where Q_m is the maximum Cu^{2+} uptake, g/L, K_L the Langmuir adsorption constant, L/mg.

3.2.2. Freundlich model

The empirical Freundlich isotherm Fig.15 is based on the equilibrium relationship between heterogeneous surfaces. This isotherm is derived from the assumption that the adsorption sites are distributed exponentially with respect to the heat of adsorption. The logarithmic linear form of Freundlich isotherm may be represented as follows:

$$\text{Log } q_e = \log K_F + (1/n_F) \log C_e \tag{15}$$

where K_F (L/g) and $1/n_F$ are the Freundlich constants indicating the sorption capacity and sorption intensity, respectively, the present data gave $1/n_F = -0.259$ and $K_F = 146.5$ (L/g), the –ve sign means the adsorption is unfavorable at high adsorbate concentration.

Figs. 14, 15 show that Langmuir adsorption isotherm gives a better data fitting than Freundlich isotherm (as shown by the high value of R²). The applicability of the Langmuir isotherm to the present data is consistent with the fact that metal ion removal from solution takes place through a chemical reaction between Cu^{2+} ions and cation exchange resin and not through physical adsorption

4. Conclusions

The present results have shown that a rotating fixed bed of ion exchange resin can form the basis of a high space time yield reactor suitable for heavy metal from waste solutions.

Within the present range of conditions Cu^{2+} removal by the rotating packed bed of ion exchange resin was found to take place through diffusion controlled first order reaction. The value of the activation energy obtained in the present work (2.19 kcal/mol) confirms the diffusion controlled mechanism of Cu^{2+} removal by the rotating fixed bed.

The mass transfer data were correlated by the following dimensionaless equation:

$$Sh = 1.235*Sc^{0.33} Re^{0.523}$$

This equation can be used in practice to design (scale up) and operate the present batch reactor. The present work revealed that the data fit the Langmuir isotherm better than the Freundlich isotherm.

The copper ion removal efficiency is highest in solutions of initial pH of 5 and decreases with increasing Cu^{2+} concentration. Increasing the amount of resin, solution temperature, and contact time increases the percentage removal of Cu^{2+}.

Future studies should include testing the effect of superimposed axial flow on the performance characteristics of the present reactor in the continuous mode..

Symbols

a — Constant
C_o — Initial concentration of copper ion (ppm)
C_e — Concentration of copper ion at equilibrium (ppm)
C_i — Interfacial concentration of copper ion at equilibrium (ppm)
C — Concentration of copper ion at any time (ppm)
E — Activation energy (kcal mol^{-1})
d — Characteristic length (basket diameter) (cm)
dp — Particle average diameter (cm)
D — Diffusivity ($cm^2 s^{-1}$)
K — Rate constant of adsorption (cm s^{-1})
K_F — Freundlich isotherm adsorption coefficient (l g^{-1})
K_L — Langmuir isotherm parameter (l g^{-1})
n_f — Freundlich isotherm constant (–)
N — Rotation speed (rps)
Q_m — Langmuir isotherm parameter (g/L)
q_e — Equilibrium mass adsorbed heavy metal per unit resin (mg/g)
R — The gas constant ($Jmol^{-1} K^{-1}$)
Re — Reynold number = $\rho N d^2/\mu$ (dimensionless)
Sh — Sherwood number = Kd/D (dimensionless)
Sc — Schmidt number = $\mu\rho/D$ (dimensionless)
t — Time (min)
T — Absolute temperature (K)
V — Volume of the solution (liter)
W — Dry weight of the ion-exchange resin (g)

Greek

ρ — Density of the solution (g/cm^3)
μ — Viscosity of the solution ($g\ cm^{-1} s^{-1}$)
δ — The diffusion layer thickness (cm)

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